Materials Chemistry and Physics 172 (2016) 26–31

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Preparation of Ce-doped colloidal SiO_2 composite abrasives and their chemical mechanical polishing behavior on sapphire substrates

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HIGHLIGHTS

- Novel Ce-doped colloidal SiO_2 composite abrasives were prepared.
- The chemical mechanical polishing (CMP) performances of the composite abrasives on sapphire substrate were investigated.
- Novel composite abrasives show excellent polishing characteristics comparison with pure colloidal SiO_2 abrasive.
- We explore and report the acting mechanism of composite abrasives to sapphire CMP.

ARTICLE INFO

Article history:
Received 20 May 2015
Received in revised form
30 October 2015
Accepted 19 December 2015
Available online 6 January 2016

Keywords:
Composite materials
Chemical synthesis
Surfaces
X-ray photo-emission spectroscopy (XPS)

ABSTRACT

Chemical mechanical polishing (CMP) has become a widely accepted global planarization technology. Abrasive is one of key elements during CMP process. In order to enhance removal rate and improve surface quality of sapphire substrate, a series of novel Ce-doped colloidal SiO_2 composite abrasives were prepared by chemical co-precipitation method. The CMP performances of the Ce-doped colloidal SiO_2 composite abrasives on sapphire substrate were investigated by using UNIPOL-1502 polishing equipment. The analyses on the surface of polished sapphire substrate indicate that slurries containing the Ce-doped colloidal SiO_2 composite abrasives exhibit lower surface roughness, higher material removal rate than that of pure colloidal SiO_2 abrasive under the same testing conditions. Furthermore, the acting mechanism of the Ce-doped colloidal silica in sapphire CMP was investigated. X-ray photoelectron spectroscopy analysis shows that solid-state chemical reactions between Ce-doped silica abrasives and sapphire surface occur during CMP process, which can promote the chemical effect in CMP and lead to the improvement of material removing rate.

1. Introduction

Sapphire, single crystal form of a-alumina, is widely used in a variety of modern high-technology applications because of its excellent optical, chemical and mechanical properties such as high hardness, great thermal stability, chemical inertness, especially used as a substrate material in the photoelectronic, microelectronic and semiconductor industries, ranging from optical windows, read/write laser sources to GaN-based light emitting diodes (LEDs), etc [1–6]. In all of these applications, the surface flatness of sapphire substrate is a key factor that influences its performances. Undoubtedly, its planarization machining is very important [7]. However, the intrinsic nature of sapphire (great hardness and chemical inertness) poses great challenges to such machining. In order to achieve the above mentioned surface quality of sapphire, several polishing technologies are put forward by researchers [8]. At present, chemical mechanical polishing (CMP) is the only widely used global planarization technology in the manufacturing of semiconductor and sapphire substrates [9–12].

In CMP, abrasives are one of key influencing factors on the polished surface quality. The sizes and distribution, dispersibility, hardness and species of abrasive are crucial for a desired CMP performance [13]. In order to improve surface planarization and material removing rate (MRR) of sapphire, several polishing abrasives have been studied. Zhu et al. [14] have studied the effect of abrasive hardness on the CMP of (0001) plane sapphire. They found that, hard abrasives (such as monocrystalline and polycrystalline

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http://dx.doi.org/10.1016/j.matchemphys.2015.12.026
0254-0584/

diamond, α- Al_2O_3 and γ-Al_2O_3) can improve MRR of sapphire, but surface arithmetic average surface roughness (Ra) which is measured at various points on the actual contour of the absolute value of the distance to the baseline of the arithmetic mean within a sampling length is higher. Nevertheless, using silica sol as polishing abrasives for sapphire can get lower Ra, but MRR of sapphire is lower. Hu. et al. [15] have researched the CMP of sapphire wafers with boron carbide and colloidal silica abrasives. They found that, B_4C abrasive eliminated the uniformity in thickness within sapphire wafer, but the colloidal silica can achieve a nanoscale flatness of sapphire surface. Xiong et al. [16] adopted silicon carbide, alumina and silica sol as polishing abrasives for CMP of sapphire. The results show that, under the same condition, Ra of sapphire polished with silica sol abrasive is lower than that with silicon carbide and alumina abrasives. In general, silica sol is widely regarded as ideal polishing abrasive for sapphire CMP since it can get better surface quality [17,18]. However, MRR of sapphire with colloidal silica as polishing abrasive is still lower and needs to be improved.

Currently, silica-based composite particles as CMP abrasives capture increasing attention, and chemical modifying of silica abrasive has been proved to be an effective way to improving its polishing performances in CMP. Zhao [19] synthesized SiO_2/CeO_2 abrasive for CMP of oxide wafer and Lei [20] developed porous Fe_2O_3/SiO_2 abrasive for CMP of hard disk substrate. Zhang et al. [21] prepared silica composite abrasive by coating with a layer of polystyrene. The silica-based composite abrasives resulted in both high planarization efficiency and good planarization quality. Up to date, preparation of silica-based composite abrasive for sapphire CMP has seldom been reported. In this paper, Ce-doped colloidal SiO_2 composite abrasives were prepared and their CMP performances on sapphire substrates were investigated.

2. Experimental

2.1. Chemicals

Chemicals used in the synthesis were ceric ammonium nitrate ($(NH_4)_2Ce(NO_3)_6$), ammonia($NH_3 \cdot H_2O$), carbamide($CO(NH_2)_2$), colloidal SiO_2, sodium hexametaphosphate and deionized(DI) water.

2.2. Preparation of material

2.2.1. Preparation of Ce-doped colloidal SiO_2 composite abrasives

Synthesis of Ce-doped colloidal SiO_2 abrasives with different CeO_2 contents: 10 wt. % crystal silica seed solution were heated to 100 °C in a four-neck flask. A certain amount of 2.5 wt.% active silicic acid solution, 0.4wt.% $(NH_4)_2Ce(NO_3)_6$ solution, 0.05 wt.% $CO(NH_2)_2$ solution and 1.5 wt. % ammonia were simultaneously added in drops into the crystal silica seed solution in the four-neck flask under continuously stirring. The liquid volume in the four-neck flask was kept constant by controlling evaporating speed of water and feeding speed of the solutions. The reaction liquid was keeping at PH of 9.0–11.0 by controlling the dropping velocity of ammonia. The solid concentration of 10 wt. % colloidal SiO_2 composite abrasive solution was obtained after the end of reaction.

In above synthesis process of composite abrasives, the cerium was doped into colloidal SiO_2 abrasives in the form of cerium oxide since the reaction liquid is alkali (At pH = 9–11, $Ce^{4+} + 4OH^- \rightarrow CeO_2 + 2H_2O$). By adding different amounts of 0.4wt. % $(NH_4)_2Ce(NO_3)_6$ solution versus crystal silica seed solution, Ce-doped colloidal SiO_2 composite abrasives containing 0.5 wt %, 1.0 wt % and 1.5 wt % CeO_2 (mass ratios CeO_2 of vs SiO_2) were obtained.

The pure colloidal SiO_2 abrasive was prepared by the same method mentioned above except adding $(NH_4)_2Ce(NO_3)_6$ solution.

2.2.2. Preparation of polishing slurries

Preparation of polishing slurries: Before polishing, 10 wt. % prepared colloidal solutions were diluted to solid concentration of 6 wt. %, then 3.0 wt. % sodium hexametaphosphate as dispersant were added into the solution. Finally, the above-mentioned mixtures were filtrated with a 10 μm pore filter to obtain polishing slurries. The pH of prepared polishing slurries is about 10.5.

2.3. Characterization of Ce-doped colloidal SiO_2 composite abrasives

The composite abrasives were characterized by means of Mastersizer 2000 instrument, scanning electron microscope (SEM), Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) and X-ray photoelectron spectroscopy (XPS).

Elementary analysis of the sample was measured by Model 2100 Trift II time-of-flight secondary ion mass spectroscopy (TOF-SIMS).

SEM analyses were accomplished using a JEOL JSM-6700F field emission scanning electron microscope with voltage of 10 kV.

The filtrate of Ce-doped colloidal silicon dioxide after polishing was characterized by the inductively coupled plasma-atomic emission spectrometry (ICP-AES, PERKINE 7300DV).

XPS spectrum was obtained by ESCALAB 250Xi electron spectrometer using a focused monochromatized Al Kα radiation ($h\upsilon = 1486.6$ eV). The binding energy of C 1s (283.35ev) was used as reference.

2.4. Polishing test

Polishing tests were conducted by using a UNIPOL-1502 polishing equipment (Shenyang Kejing instrument, Co. LTD, China). The polishing pad was a Rodel porous polyurethane pad. The slurries supplying rate is 180 ml/min. The parameters of polishing were down force of 6 kg, plate rotating speed of 60 rpm and conditioning time of 2 h. Work pieces were φ50.8 mm × 0.45 mm α-Al_2O_3 sapphire substrates with c (0001) orientation. The average roughness (Ra) and of the sapphire substrates are about 2.535 nm. After polishing, the Sapphire substrates were washed with ultrasonic in a cleaning solution containing 0.5 wt.% surfactant in deionized(DI) water. Finally, they were dried by a multifunctional drying system.

Ra was measured by an Ambios XI-100 surface profiler (Ambios Technology Corp., U.S.A) with the resolution of 0.1 Å and the measuring area of 500 μm × 500 μm. The depth of focus is 3.0 μm, the working distance is 7.4 mm, and the numerical aperture is 0.30. The mass of the sapphire substrate was measured by a AL104 analytical balance with accuracy of 0.1 mg (METTLER TOLEDO, Switzerland). The material removal rate was calculated by formula. (1).

$$MRR = \frac{\Delta m \times 10^6}{\rho \pi R^2 \tau} \quad (1)$$

where:

MRR-material removal rate, μm/h
Δm-mass of material removed of substrates before and after polishing, g
R-radius of sapphire substrate, mm
τ-polishing time, h (in the test, τ = 2 h)
ρ-density, g/cm^3 (density of sapphire substrate, ρ = 3.98 g/cm^3).

3. Result and discussion

3.1. Characterization of Ce-doped colloidal SiO_2 composite abrasives

Fig. 1 is SEM spectra of pure colloidal SiO_2 abrasive and Ce-doped colloidal SiO_2 composite abrasives. The result shows that, by comparison with the pure colloidal SiO_2 abrasive (0 wt % of CeO_2), the particle sizes of Ce-doped colloidal SiO_2 composite abrasives are almost no change. And the prepared Ce-doped colloidal SiO_2 abrasives have inerratic spherical shape and excellent dispersibility.

TOF-SIMS is an effective means for element analysis because of its high sensitivity and mass and space resolution. In order to analyze the composition of Ce-doped colloidal SiO_2 composite abrasives, elementary analysis of the samples of the pure colloidal SiO_2 abrasive and the composite abrasive were measured by TOF-SIMS. The results are shown in Fig. 2, horizontal axis is ion mass (amu), and vertical axis is number of ions counted. It is seen that silicon and cerium are present in the composite abrasive in Fig. 2b while cerium is almost not present for pure colloidal SiO_2 in Fig. 2a,

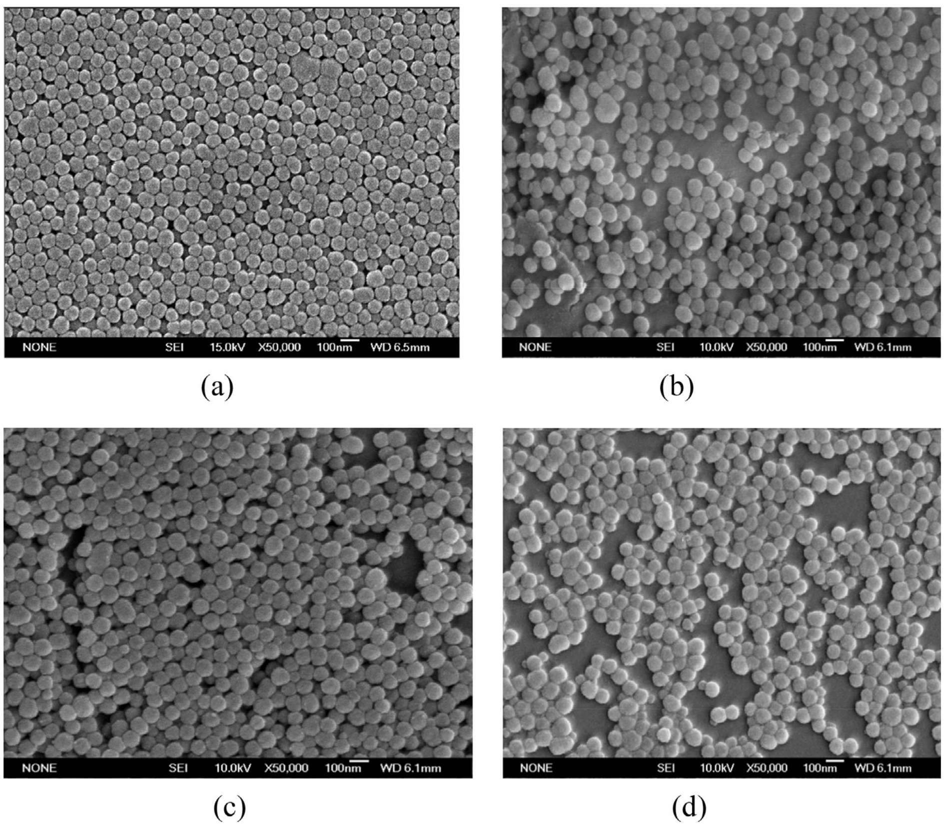

Fig. 1. SEM spectra of Ce-doped colloidal SiO_2 composite abrasives with different CeO_2 contents (a) 0 wt.% of CeO_2, (b) 0.5 wt.% of CeO_2, (c) 1.0 wt.% of CeO_2, (d) 1.5 wt. % of CeO_2.

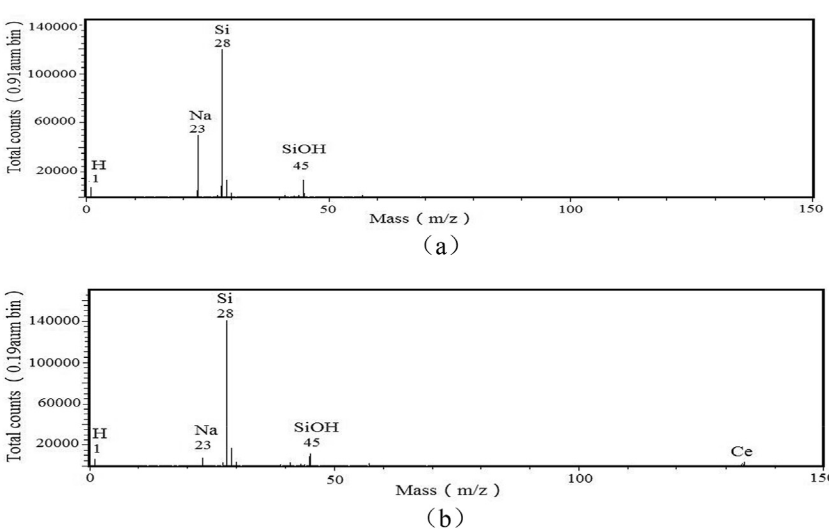

Fig. 2. TOF-SIMS spectra of pure colloidal SiO_2 abrasive (a) and Ce-doped colloidal SiO_2 abrasive with 1.5 wt. % CeO_2 (b).

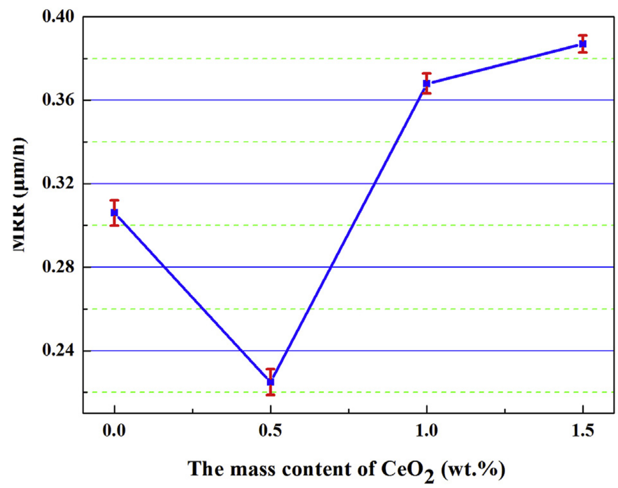

Fig. 3. Effect of the mass content of CeO_2 on material removal rate (Red line is error bar that is ± 1.96 standard deviation and represents a description of 95% confident of which the mean represents the true MRR value) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.).

Table 1
The experiment error analysis data of three polishing tests.

The mass of CeO_2 (wt.%)	0	0.5	1	1.5
1	0.318	0.230	0.359	0.379
2	0.298	0.235	0.379	0.396
3	0.302	0.210	0.366	0.386
Mean	0.306	0.225	0.368	0.387
Standard deviation	0.0106	0.0108	0.0083	0.0070

which illustrate that cerium element was successfully introduced into colloidal SiO_2 composite abrasives.

3.2. CMP performances of the Ce-doped colloidal SiO_2 composite abrasives

In order to investigate the difference in polishing performances between the prepared Ce-doped colloidal SiO_2 composite abrasives and pure colloidal SiO_2 abrasive, the MRR and Ra of polished sapphire substrate surfaces were analyzed. Fig. 3 and Table 1 reveal the effects of different CeO_2 contents upon the MRR of the sapphire. It can be seen that, with increasing of the mass content of CeO_2, the MRR of sapphire substrates decrease at first and then increase gradually. The MRR of sapphire substrates polished in pure colloidal SiO_2 slurry is 0.306 μm/h, while the MRRs of CeO_2-doped composite abrasives with 1.0% CeO_2 and 1.5% CeO_2 give 0.368 μm/h and 0.387 μm/h, respectively. The lower MRR of CeO_2-doped composite abrasive with 0.5% CeO_2 content may be due to the weaker chemical action and lower hardness of CeO_2-doped composite abrasive at lower CeO_2 doping content. In other words, the prepared Ce-doped colloidal SiO_2 composite abrasives at a suitable CeO_2 content possess higher MRR than pure colloidal SiO_2 abrasive.

In addition, to get more details about the differences in polishing performances between the composite abrasives and pure colloidal SiO_2 abrasive, the Ra values and topographical micrographs of polished sapphire surfaces were obtained by an AMBIOS TECHNOLOGY XI-100 Optical Surface Profiler. As displayed in Fig. 4, a rough surface is observed on the surface before polishing and the roughness is as high as 2.535 nm (Fig. 4a). After polishing by the pure colloidal SiO_2, the surface of the sapphire substrate is less rough and has a Ra of 2.197 nm (Fig. 4b). And, it is found that the prepared composite abrasive with 1.5% CeO_2 doping content gives

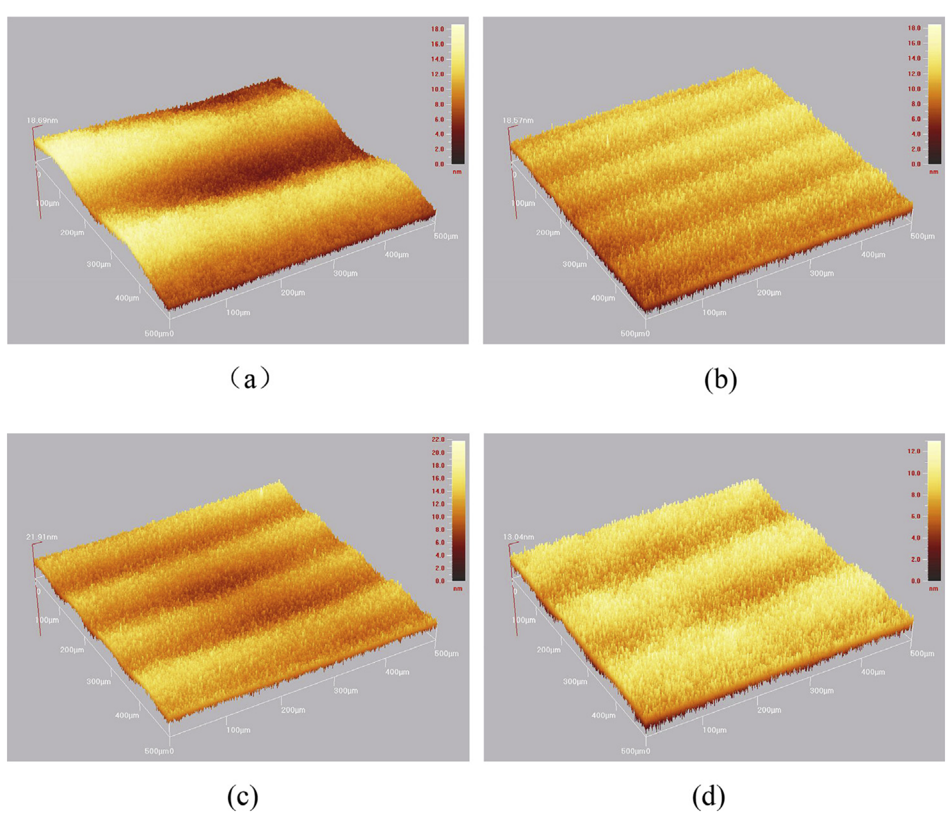

Fig. 4. Surface profiles of sapphire substrates (a) before polishing, Ra = 2.535 nm; (b) polished by pure colloidal SiO_2, Ra = 2.197 nm; (c) polished by Ce-doped colloidal SiO_2 containing 0.5wt. % CeO_2, Ra = 1.879 nm; (d) polished by Ce-doped colloidal SiO_2 containing 1.5wt. % CeO_2, Ra = 1.376 nm.

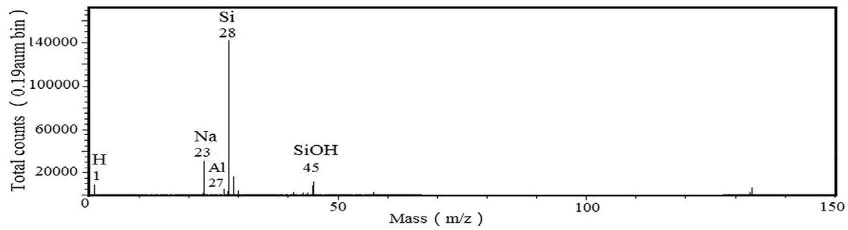

Fig. 5. TOF-SIMS spectra of the slurry after polishing.

Table 2
ICP-AES consequence of Ce-doped colloidal SiO_2 polishing slurry after polishing.

Element content (mg/L)	Al	Ce
filtrate	0.2	0
polishing slurry	226.24	798.08

Ra of 1.376 nm (Fig. 4d). The lower Ra value means the higher surface planarization. In other words, the prepared Ce-doped colloidal SiO_2 composite abrasives possess higher surface planarization than pure colloidal SiO_2 abrasive.

3.3. Mechanism discussion

CMP is a process of chemical action and mechanical action mutual coordination. In order to explore the acting mechanism of Ce-doped colloidal SiO_2 composite abrasives to sapphire CMP, the slurry containing Ce-doped colloidal SiO_2 composite abrasive was analyzed by TOF-SIMS, ICP-AES and XPS.

The slurry containing Ce-doped colloidal SiO_2 composite abrasive with 1.5 wt. % CeO_2 after polishing was dried and was characterized by TOF-SIMS.

The slurry containing Ce-doped colloidal SiO_2 composite abrasive with 1.5 wt. % CeO_2 after polishing was centrifuged. The residual of the solid precipitation was washed with deionized water for many times and dried at normal temperature, and then analyzed by XPS. The filtrate which is supernatant of centrifuged the slurry containing Ce-doped colloidal SiO_2 composite abrasive with 1.5 wt.% CeO_2 after polishing was characterized by ICP-AES.

Fig. 5 shows result of TOF–SIMS, Al ion is present in the slurry after polishing. It means that aluminum had been introduced into slurry. Table 2 shows results of ICP-AES. The result indicates that

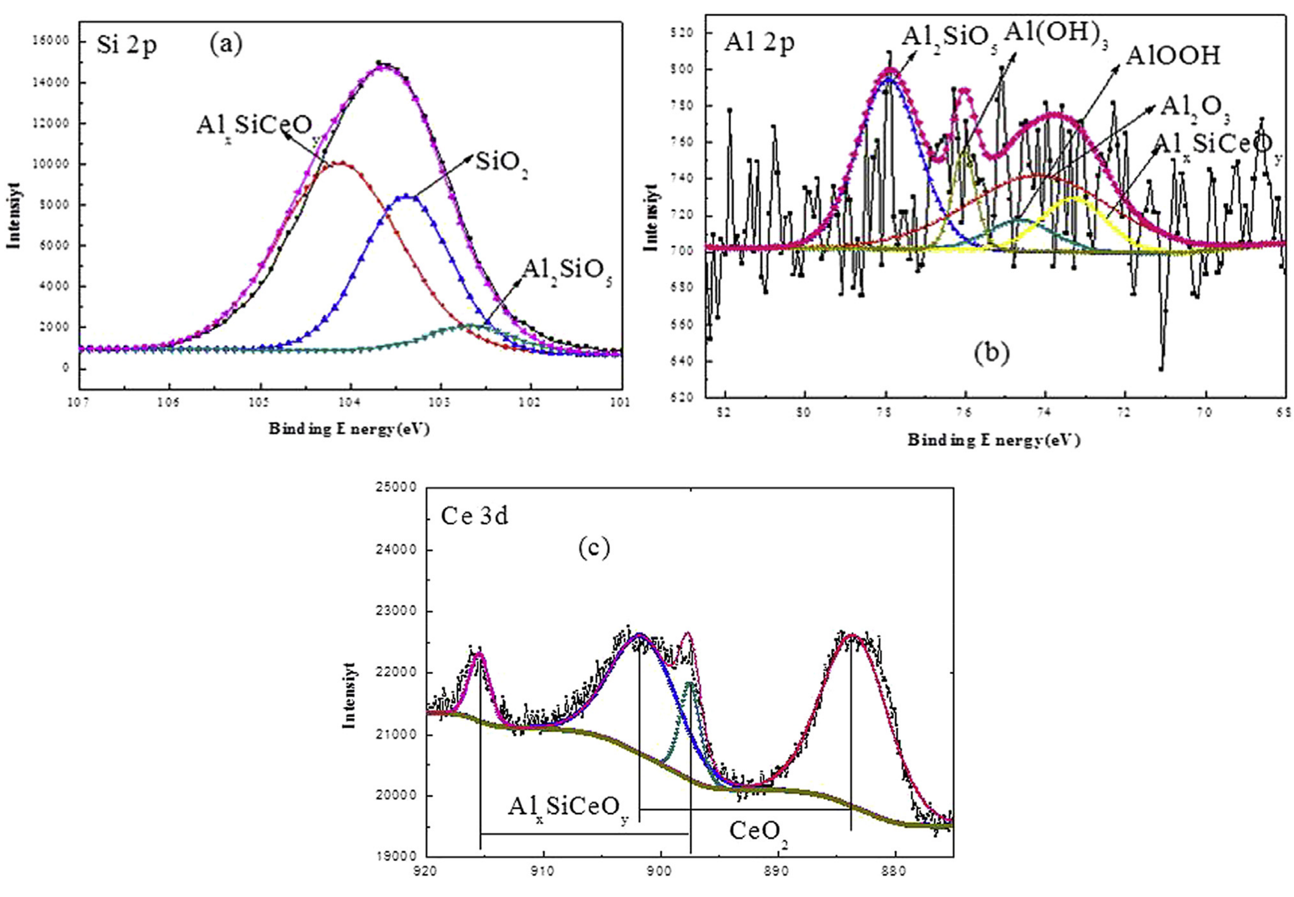

Fig. 6. The XPS narrow scan spectra results of element Si (a), element Al (b) and element Ce (c) of Ce-doped colloidal SiO_2 composite abrasive containing 1.5 wt% CeO_2 after polishing sapphire (black lines are experiment results and magenta lines are fitting results).

Table 3
Binding energy of Si2p.

Chemical state	Binding Energy(eV)
Al_2SiO_5	102.68
Al_xSiCeO_y	103.25
SiO_2 (gel)	104.09

Table 4
Binding energy of Al2p.

Chemical state	Binding Energy(eV)
Al_2O_3 (sapphire)	73.4
$Al(OH)_3$	75.94
AlOOH	74.6
Al_2SiO_5	74.9
Al_xSiCeO_y	73.04

Table 5
Binding energy of $Ce3d_{5/2}$.

Chemical state	Binding Energy(eV)
CeO_2	882.38
Al_xSiCeO_y	897.48

the aluminum element can not be observed. In other word, aluminum with the form of free ions (AlO_2^- or Al^{3+}) is not existent in polished slurry. The consequence illustrates that all the element aluminum polished from sapphire surface should exist in precipitation of analyzed slurry.

In order to further reveal the mechanism of Ce-doped colloidal SiO_2 abrasives on sapphire CMP, XPS analyses of the residuum of the solid precipitation of polishing slurry was conducted. Fig. 6 shows the XPS narrow scan spectra results of element Si (a), element Al (b) and element Ce (c) of Ce-doped colloidal SiO_2 abrasive with 1.5 wt% CeO_2 after polishing, and the binding energy of different material is presented in Tables 3–5. In Fig. 6, the black line shows the real total intensity measured by XPS test, and the magenta line shows the total intensity after curve fitting by using the Thermo Avantage software. The closer between the real result (black line) and the fitting result (magenta line) will lead to more accurate analysis results. The peak at the binding energy (BE) of 104.09 eV (Fig. 6a) corresponds to Si2p in SiO_2 (gel) state and the peak at the binding energy (BE) of 73.4 eV (Al2p) corresponds to Al_2O_3 sapphire (Fig. 6b). And the peak at the binding energy (BE) of 882.38 eV (Fig. 6c) corresponds to $Ce3d_{5/2}$ as CeO_2. Fig. 6a, b reveals that the peak at the binding energy (BE) of 102.68 eV corresponds to Si2p in Al_2SiO_5 state as well as the peak at the binding energy (BE) of 74.9 eV corresponds to Al2p in Al_2SiO_5 state. And the peak at the binding energy (BE) of 74.6 eV corresponds to Al2p in AlOOH state. The consequence illustrates solid-state chemical transformation react between SiO_2 and AlOOH. At the pH of 10.5, this change can be expressed in Equations (2) and (3) [22].

$$Al_2O_3 + H_2O \rightarrow 2AlOOH \tag{2}$$

$$2AlOOH + SiO_2 \rightarrow Al_2SiO_5 + H_2O \tag{3}$$

In addition, Fig. 6c, the peak at the binding energy (BE) of 897.48 eV (Fig. 6c) corresponds to $Ce3d_{5/2}$ as Al_xSiCeO_y as well as the peak at the binding energy (BE) of 103.25 eV (Fig. 6a) corresponds to Si2p in Al_xSiCeO_y, which may be the compound of Al, Si and Ce oxide. This change can be expressed in Equation (4).

$$CeO_2 + SiO_2 + Al_2O_3 \rightarrow Al_xSiCeO_y \tag{4}$$

To sum up, the consequence illustrates solid-state chemical reaction occurs during the polishing process. The improvements of sapphire CMP may be attributed to the solid-chemical reaction between sapphire surface and Ce-doped colloidal SiO_2 composite abrasives. Simultaneously, the softer CeO_2 introduced into colloidal SiO_2 abrasive leads to little scratches on the sapphire surface comparing with pure colloidal SiO_2 abrasive.

4. Conclusions

Novel Ce-doped colloidal SiO_2 composite abrasives were prepared. The results show that the prepared Ce-doped colloidal SiO_2 composite abrasives have good dispersibility and inerratic spherical shape. The slurries containing Ce-doped colloidal SiO_2 composite abrasives exhibit lower Ra and higher MRR than that containing pure SiO_2 abrasive in the CMP of sapphire substrates under the same conditions. The improvements of sapphire CMP may be attributed to its solid-chemical reaction between sapphire surface and Ce-doped colloidal SiO_2 composite abrasives. Meanwhile, softer CeO_2 doping and good dispersibility of the composite abrasives endue their higher surface planarization in sapphire CMP.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant No. 51475279, No. 91323302). Research Fund for Doctoral Program of Higher Education of China (Grant No. 20123108110016).

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